A CRYOSTAT FOR NEUTRON DIFFRACTION STUDIES

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Submitted to JETP editor October 10, 1959

J. Exptl. Theoret. Phys. (U.S.S.R.) **38**, 806-808 (March, 1960)

A low temperature (including helium temperatures) cryostat is described for studying scattering of thermal neutrons by polycrystalline specimens.

WE have constructed an apparatus for studying the magnetic properties of antiferromagnetics at low temperatures,[1,2] in which neutron diffraction can be investigated over a wide range of Bragg angles. As the requirements of accuracy in interpreting the diffraction pattern are high, we took account of many reflections and high orders. It was therefore experimentally desirable to be able to measure the scattering up to angles $\Theta_B = 45°$, i.e., for rotation of the detector through 90°. The optimum rate for the automatic rotation of our detector around the specimen is 1° in 3.5 min. It follows, then, that the exposure time for recording the diffraction pattern can be 5 hours, and if the picture is taken point by point this time is doubled.

The main requirement for a cryostat for this purpose is, then, to maintain the specimen at a low temperature for a long time with, naturally, minimum background scattering produced by the cryostat itself.

The cryostat used for our measurements was constructed from the hydrogen Dewar proposed for the purpose by Kapitza in 1932.[3] Figure 1 shows a sketch of the Dewar after it had been turned into the cryostat. "Appendix" 2, of 0.2 mm wall thickness stainless steel, is attached below the inner vessel 1, which holds 5 *l* of liquid. The thin walled (0.2 mm) aluminum cup 3, containing the specimen, is attached to the base of the "appendix." The aluminum was tinned with an ultrasonic soldering iron before being soldered on. The leads to the thermometer 6 were passed to the outside through a 3 mm diameter stainless tube 4, extending through the whole cryostat, from the bottom of the appendix to a sealed glass tube 5, provided with platinum leads.

The 5-liter sphere 1 is surrounded by a copper shield 7 cooled by nitrogen fed from a 3-liter "sputnik," 9, through a copper rail 8. The aluminum foil cylinder 11 is attached to shield 7. There

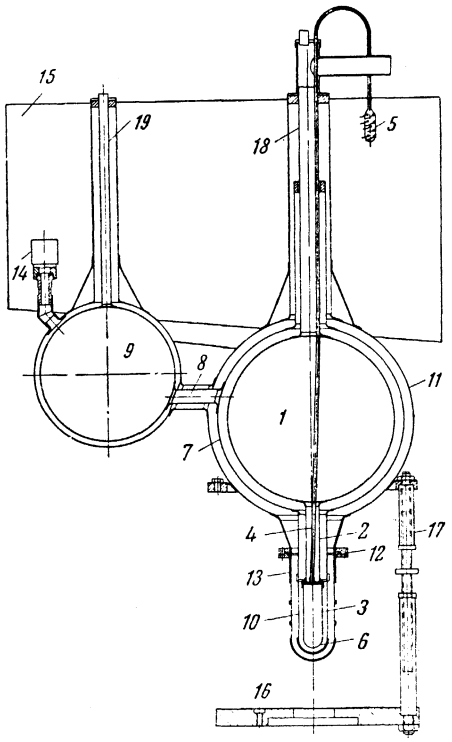

FIG. 1

is a flange connection 12 below the outer layer of the cryostat by means of which the aluminum pressure vessel 13 is attached to the outer layer with 4 mm screws through a rubber seal. This vessel is thinned down to a thickness of 0.2 mm over the specimen height, namely 60 mm, with stiffening rings 1.5 mm thick left on the surface at 20 mm intervals. The suspension tubes 18 and 19 of the vessels 1 and 9 were changed to stainless steel to reduce the heat inflow. The rubber seal, aluminum soldering and thinned-down cylinder operated without trouble after a year and half.

The vacuum space between the spheres is pumped through the sylphon tube 14, fixed to the Plexiglas shield 15, by a special vacuum system connected by a flexible metal tube. The original vacuum achieved, 2×10^{-5} mm Hg, did not deteri-

orate during operation. A series of measurements on any one specimen could be carried on for several months without further pumping. This was aided by activated charcoal at the bottom of sphere 1 and the nitrogen sphere, which on cooling absorbed the gas given off the inner surfaces of the cryostat.

During the experiments the sputnik was constantly supplied with nitrogen by an oxygen-regulated automatic filler. When assembled, the cryostat with the specimen is located at the center of the mounting of the neutron spectrometer by means of the aluminum disc 16 and the three supports 17.

When experiments are made at helium temperatures, the helium evaporated from the cryostat is collected by a pumping system attached for the purpose. Helium from a portable Dewar (see reference 4) is siphoned into the 5-liter volume kept at a constant pressure of 20 mm Hg, produced by the forevacuum pump. The pump exhaust is connected to a (2.5 m^3) rubber and a (0.5 m^3) metal gas holder. The helium is compressed at 5 m^3 per hour into a high pressure container by a compressor. The use of a rubber gas holder avoids an expensive and bulky metal holder and also allows the expermenter to carry out the whole process of filling and pumping helium without assistance.

As has been found from many experiments, the cryostat fulfilled all the requirements demanded of it. The background from the aluminum casing is 20% of the counter background and does not distort the neutron diffraction pattern of the specimen. The coolants kept for a sufficiently long time, and at the end of the diffraction measurements had to be boiled off from inside the 5-liter sphere by a special heater.

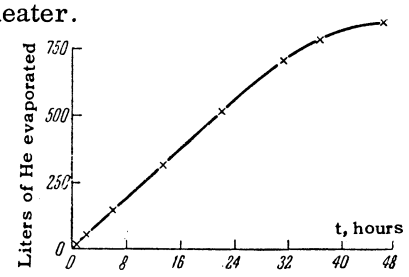

FIG. 2

The apparatus could keep a specimen at helium temperature for two days and nights. The total amount of helium used for an experiment, including the cooling, was then 7.5 l. A typical evaporation curve is shown in Fig. 2, which shows the evaporation of 1.3 l He. The rate of evaporation is at first constant (1 l of liquid is boiled off in 29 hours), and then gradually decreases up to 69 hours. This corresponds to the moment when helium only remains in the "appendix" 2. Most of the helium is, therefore, boiled off while the apparatus is cooling down, the evaporation itself being small. We have calculated that ~ 4 l of liquid He are necessary to cool the vessel.

We used an experimental thermometer obtained from Yu. V. Sharvin, prepared from coal[5]. Such thermometers, especially the resistance thermometers made by the Allen-Bradley company, show a strong temperature dependence of resistance at low temperatures. This makes them very convenient as low-temperature thermometers. They are advantageous for neutron diffraction work because of their extremely small size. A small thermometer can be placed on one end of the cylinder with the specimen and so does not affect the diffraction pattern.

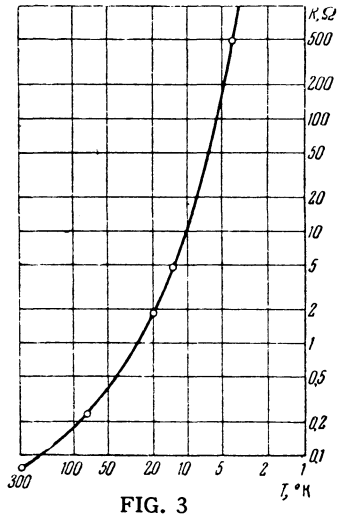

FIG. 3

Our thermometer was 1 × 2 × 3.5 mm in size and was secured with BF-2 adhesive to the bottom of the aluminum cylinder containing the specimen. Potential and current leads from it were connected to a compensated potentiometer circuit. Figure 3 shows the thermometer calibration, measured at five temperatures.

In some cases, temperature control of the specimen for long experiments was achieved by a recording potentiometer EPP-09 with 100 scale divisions for 10 mv.

I am deeply grateful to Acad. P. L. Kapitza for his constant interest in the work. I thank Yu. V. Sharvin for supplying me with an experimental thermometer model.

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Translated by R. Berman
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